

Department of Pesticide Regulation Environmental Monitoring Branch 1001 I Street Sacramento, California 95812 May 20, 2005

Study 229. Continuing Assessment of Pyrethroid Contamination of Surface Waters and Bed Sediments in High Pyrethroid-Use Regions of California

I. INTRODUCTION

Synthetic pyrethroid insecticides are applied to a variety of crops in California throughout the year. In 2003, over 280,000 pounds of pyrethroid insecticide active ingredients were applied to agricultural fields throughout the state. Six of the primary pyrethroids used in California agriculture, in order of decreasing amount applied in 2003, are permethrin, esfenvalerate, lambda-cyhalothrin, cyfluthrin, bifenthrin and cypermethrin (DPR 2005). Use of pyrethroid insecticides is expected to increase as they are utilized more commonly as replacements for organophosphate insecticides.

Due to the aquatic toxicity of the pyrethroid insecticides (Table 1), offsite movement of these compounds into surface water is of concern. Recent monitoring studies conducted in California have shown pyrethroid contamination of both surface water/suspended sediment and stream bed sediment (Weston *et al.*, 2004; Gill and Spurlock, 2004; Bacey *et al.*, 2003; Walters *et al.*, 2002; Kelley and Starner, 2004; Starner and Kelley, in preparation). Considering their high and potentially increasing use in California, reliable information regarding the environmental fate of these compounds is increasingly important.

The pyrethroid insecticides are extremely hydrophobic, with high adsorption coefficients and very low water solubility (Table 1). Due to their adsorption properties, determining the impact of pyrethroids on aquatic systems requires monitoring of suspended and bed sediments in addition to surface water (Weston *et al.*, 2004).

In 2004, the California Department of Pesticide Regulation (DPR) initiated a survey monitoring study (Starner, 2004) designed to begin assessing the extent of pyrethroid contamination of the aquatic environment in high-use regions of the state (Table 2). Four regions of high pyrethroid-use (Salinas River, Sacramento Valley/Feather River, Northern San Joaquin Valley, and Imperial Valley) were sampled three times each over a 12-month period (July 2004 – June 2005). Bed sediment and whole water samples were analyzed for pyrethroid insecticides (Starner and Kelley, in preparation). The current study is a continuation and expansion of the 2004 study, with modifications to the study design based on the results of the initial survey and recent pesticide use data (Figures 1-4).

Salinas Valley/Salinas River Region, Monterey County

Over 39,000 pounds of pyrethroid insecticide active ingredients were applied to agricultural crops in Monterey County in 2003. Use in 2001-2002 was similar (Figure 1). Of this amount, most was applied within an approximately 350 square mile area in the Salinas Valley region. This region extends approximately 50 miles along the Salinas River from Monterey Bay north of the town of Marina southeast to about King City. Much of this area is cultivated year-round, with associated intensive use of pyrethroid insecticides and other pesticides.

The high-use period in the Salinas Valley region extends from April through October; over 80% of all pyrethroid use in the region occurs during this period (Figure 5). In 2003, over 34,000 pounds of pyrethroid active ingredients were applied during this seven-month period, averaging over 4,500 pounds

Note: All pesticide use data cited are agricultural use data from DPR 2005.

applied per month. Use on lettuce, spinach, celery and artichokes accounted for 90% of the total amount applied in 2003 (Figure 6). During the low-use season, December through February, total pyrethroid applications in the region generally average less than 400 pounds per month.

The Salinas River flows into the Monterey Bay National Marine Sanctuary, and the lower river is a primary migration corridor for endangered steelhead trout (*Onchorhynchus mykiss*)(Anderson *et al.* 2003, Busby *et al.*, 1997).

Imperial Valley/Alamo River Region, Imperial County

Pyrethroid insecticides are applied throughout the year in Imperial County, with over 21,000 pounds applied in 2003. Use was similar in 2001 and 2002 (Figure 2). Throughout the county, more than 1,000 pounds of the six primary pyrethroid insecticide active ingredients were applied each month in nine out of twelve months during 2003 (Figure 7).

Virtually all agricultural use of pyrethroid insecticides in Imperial County takes place within an approximately 650 square mile area within the Imperial Valley. From the southeastern shoreline of the Salton Sea, the high use region extends east approximately 20 miles and south to the US/Mexico border. In 2003, over 20,000 pounds of the six pyrethroids were applied to agricultural crops within this 650 square mile area.

Two distinct periods of relatively high pyrethroid use occur within the Imperial Valley region (Figure 7). The highest use occurs during the fall (October and November), with applications made primarily to vegetable crops. A second period of high use occurs in late winter (normally February and March), when applications are made primarily to alfalfa. These two high use periods together account for approximately 60% of the annual pyrethroid use in the region. Additionally, use is over 1,000 pounds per month for the remainder of the year except in the warmest summer months, June through August (Figure 8). Monitoring in this region will be conducted in appropriate locations during these high use periods.

Agricultural drain water from the Imperial Valley drains into the Salton Sea, providing over 70% of the freshwater input to the Sea. The Salton Sea supports a National Wildlife Refuge and is a critical stop on the Pacific Flyway for migrating birds (California Regional Water Quality Control Board, Colorado River Basin, 2003).

Sacramento Valle y

Pyrethroid insecticides are applied throughout the Sacramento Valley (Butte, Colusa, Glenn, Placer, Sacramento, Solano, Sutter, Yolo and Yuba counties) on a variety of crops, including fruits, nuts, vegetables and rice. Nearly 25,000 pounds of the six primary active ingredients were applied in 2003 throughout the entire valley; use in 2001-2002 was similar (Figure 3).

The primary pyrethroid use period in the area occurs during May through August, with applications to peaches, tomatoes and rice accounting for about 55 percent of the total use during that period. A secondary use period occurs in spring, with applications to alfalfa (Figures 8 and 9). The Feather River region, which accounts for much of the pyrethroid use on peaches in this region, was monitored in a previous DPR study and results from that study are forthcoming (Starner and Kelley, in preparation). For the current study, monitoring in the Sacramento Valley will be conducted in appropriate locations outside of the Feather River area, during the high use periods described above.

The area encompasses the Sacramento and Feather Rivers. The Feather River is a tributary of the Sacramento River, which drains to the Sacramento Delta and to San Francisco Bay.

Northern San Joaquin Valley/San Joaquin River Region

Pyrethroid insecticides are applied throughout the eight county region of the San Joaquin Valley (Fresno, Kern, Kings, Madera, Merced, San Joaquin, Stanislaus and Tulare counties) on a variety of crops, including nuts, fruits, corn, cotton and alfalfa. Over 120,000 pounds of pyrethroid active ingredients were applied in 2003 throughout the entire valley.

While pyrethroid use is significant throughout the entire valley, the greatest potential for movement of pyrethroids to surface water bodies occurs in several localized (geographically distinct) areas of high use within the northern San Joaquin Valley. These areas include parts of San Joaquin, Stanislaus and Merced counties (Figure 4). Use in this region is highest throughout the summer months, especially in July, with additional periods of high use in January and March (Figure 9).

In this three county region, over 35,000 pounds of pyrethroid active ingredients were applied in 2003. In 2003, approximately one-third of all pyrethroid use in this region (over 12,000 pounds) occurred in July, primarily to almonds but with significant use also on tomatoes and corn (Figure 10). Additionally, over 2,000 pounds were applied to almonds in January, and nearly 3,500 pounds to alfalfa in March. Use was similar in 2001 and 2002. Monitoring in this region will be conducted in appropriate locations during these high use periods.

The area encompasses the San Joaquin River, which drains to the delta and San Francisco Bay, and several major tributaries of the San Joaquin, including the Stanislaus, Tuolumne and Merced Rivers.

II. OBJECTIVE

The primary objective of this study is to more fully characterize the extent of pyrethroid insecticide contamination of surface waters in four high-use regions of California.

The secondary objective is to acquire additional surface water monitoring data for organophosphate (OP) insecticides in those same regions. In general, the high pyrethroid use periods in the four regions coincide with high OP use (DPR 2005). Ongoing collection of diazinon and chlorpyrifos surface water data is needed because these organophosphate insecticides have been placed into re-evaluation by DPR (DPR 2003a and 2004b).

Results will be used to aid in the development of priorities for future monitoring and/or mitigation efforts.

III. PERSONNEL

The study will be conducted by staff from the Environmental Monitoring Branch, Surface Water Protection Program, under the general direction of Kean S. Goh, PhD., Agricultural Program Supervisor IV. Key personnel are listed below:

Project Leader: Keith Starner Field Coordinator: Kevin Kelley

Senior Scientist: Frank Spurlock, Ph.D. Laboratory Liaison: Carissa Ganapathy

Chemists: California Department of Food and Agriculture, Center for Analytical Chemistry

Staff Chemists

Questions concerning this monitoring project should be directed to Keith Starner at (916) 324-4167.

IV. STUDY PLAN

The study is designed to include sampling of one region (Salinas River region) approximately monthly throughout a twelve-month period, during both low and high pyrethroid use periods. The remaining three regions (Imperial Valley, Sacramento Valley and northern San Joaquin Valley) will be sampled two to three times during their respective high-use periods. This will allow for a more thorough characterization of the Salinas River region while providing additional information on the remaining three regions. If warranted, more intensive sampling of these regions may be included in future monitoring efforts.

Within each region, four to six sites will be sampled in each region at each sampling interval. Locations of individual sites within a region will be determined based on the historical pyrethroid use patterns, proximity to the water bodies of interest, and previous pyrethroid monitoring data, where available. Where appropriate, sampling site locations within each region will be selected and/or modified based in part on results of the 2004 survey study.

Site selection will follow the general guidelines in Standard Operating Procedure (SOP) FSWA002.00 (Bennett, 1997) where applicable.

Sampling will commence in July 2005 and continue through June 2006.

V. SAMPLING METHODS

At each sampling site, one sediment sample will be collected into a pint Mason jar for pyrethroid analysis. An additional sediment sample will be collected for total organic carbon (TOC) analysis. Where fine sediment is present on the bed surface, only the top 1 - 2 cm of the sediment column will be collected. Where possible, a trowel will be used to collect the samples by gently scraping the top layer of the sediment column. If necessary, a core tube will be used, retaining only the top 1 cm from each core and taking care to minimize disturbance of the top sediment layer during the sampling process. Where possible, sediment samples will be collected from a subregion of the selected sampling site most likely to result in collection of recently deposited sediment.

Sediment samples will be transported on wet ice and transferred as soon as practicable to frozen storage at 0°C until extraction for chemical analysis.

At each sampling site, two surface water grab samples, one for pyrethroid analysis and one for organophosphate analysis, will be collected directly into separate 1-liter amber glass bottles. An additional surface water grab sample will be collected for total suspended solids (TSS) analysis. Grab samples will be collected as close to center channel as possible using a grab pole consisting of a glass bottle at the end of an extendable pole, or other sampling equipment designed to collect a sample directly into a 1-liter glass bottle. Samples will not be transferred from the original sample bottles until analysis at the lab. Amber bottles will be sealed with Teflon-lined lids and samples will be transported and stored on wet ice or refrigerated at 4°C until extraction for chemical analysis.

Dissolved oxygen, pH, specific conductivity, and water temperature will be measured *in situ* at each site during each sampling period. Flow data will be collected using a digital flow meter.

VI. CHEMICAL ANALYSIS

Chemical analysis will be performed by the California Department of Food and Agriculture's Center for Analytical Chemistry. Analytical method titles and reporting limits for this study are given in Table 3.

Details of the chemical analysis methods will be provided in the final report. Quality control will be conducted in accordance with Standard Operating Procedure QAQC001.00 (Segawa, 1995). Ten percent of the total number of analyses will consist of field blanks and blind spikes, to be submitted to the laboratory with field samples.

All sediment samples and whole water samples collected for pyrethroid analysis will be analyzed for bifenthrin, cyfluthrin, cypermethrin, esfenvalerate, lambda-cyhalothrin, and permethrin. For pyrethroid water analyses, the whole sample, including any suspended sediment, will be extracted in the sample bottle (*in toto*) and the pyrethroid residues will be reported on a whole sample basis (water plus suspended sediment). Replicate whole water samples will be collected at each sampling event and analyzed to determine suspended sediment concentration. Whole water samples collected for organophosphate insecticide analysis will be analyzed for a suite of organophosphates, listed in Table 3.

VII. DATA ANALYSIS

Concentrations of pesticides in water will be reported as micrograms per liter ($\mu g/L$) or nanograms per liter (ng/L). Concentrations of pesticides in sediment will be reported as micrograms per kilogram ($\mu g/kg$).

Resulting data will be analyzed and reported as appropriate, potentially including the following:

Comparison of analytical concentrations to concurrent use data, toxicity data, and Water Quality Criteria (CDFG 2000);

Spatial analysis of the resulting data using Geographic Information System (GIS) software in order to identify correlations between observed pesticide concentrations and region-specific geographical features such as climate, soil type, cropping patterns and agricultural practices.

Dissolved and sorbed pyrethroid concentration in whole water pyrethroid samples will be estimated (Spurlock, 2003).

VIII. TIMETABLE

Field Sampling: July 2005 through June 2006 Chemical Analysis: July 2005 through September 2006

Final Report: April 2007

IX. BUDGET

Primary Analysis		Cost (@ \$	300/sa	mple)
Pyrethroid screen, sediment	1 rep x 6 sites x 12 events =	72 samples	=	21,600
	1 rep x 4 sites x 6 events =	24 samples	=	7,200
Pyrethroid screen, water	1 rep x 6 sites x 12 events $=$	72 samples	=	21,600
	1 rep x 4 sites x 6 events =	24 samples	=	7,200
Organophosphate screen (incl. diazinon)	1 rep x 6 sites x 12 events $=$	72 samples	=	21,600
	1 rep x 4 sites x 6 events =	24 samples	=	7,200
Quality Control				
Blind spikes		15 samples	=	4,500
Field blanks		15 samples	=	4,500
Total				95,400

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Table 1. Pyrethroid Physical and Toxicological Characteristics

Pesticide	K _{OC}	Solubility (mg/l)	Half-life Soil (days, aerobic/anaerobic)	Hydrolytic (pH 7) Half-life (days)	Sediment Toxicity A LC ₅₀ Hyalella azteca (µg/g OC)	Aquatic Toxicity B LC ₅₀ Daphnia Magna (ppb)
Bifenthrin	237,000	1.4e ⁻⁵	96/425	Stable	0.52	1.6
Cyfluthrin	124,000	2.3e ⁻³	12/34	183	1.08	0.16
Cypermethrin	310,000	4.0e ⁻³	28/55	274	0.18-0.6 ^C	1.25
Esfenvalerate	215,000 ^B	6.0e ⁻³	39/94	Stable	1.54	0.24
lambda- cyhalothrin	326,000	5.0e ⁻³	43/na ^D	Stable	0.45	0.23
Permethrin	277,000	5.5e ⁻³	40/197	Stable	10.83	0.075

Source: Laskowski, 2002 except as indicted.

A. Amweg et al., 2005. Data are median values, in μ g/g OC (organic carbon).

B.DPR 2003b.

C. Maund et al., 1998.

D. na = not available.

Table 2. Pyrethroid use in high use counties, by active ingredient (ai), 2003.

County	Pounds ai A ₁	pplied, 2003					
					Lambda-		Total by
	Bifenthrin	Cyfluthrin	Cypermethrin	Esfenvalerate	Cyhalothrin	Permethrin	County
Butte	64	18	385	947	287	1688	3388
Colusa	103	93	231	385	350	1968	3130
Glenn	221	37	347	668	510	1774	3559
Imperial	399	2974	4693	2829	1932	8397	21224
Merced	1328	1533	80	1584	1096	5876	11497
Monterey	1225	9	6209	3101	2271	26772	39587
Sacramento	50	0	0	253	173	1179	1655
San Joaquin	485	74	22	3231	627	6759	11199
Stanislaus	1386	301	201	4139	899	5386	12312
Yolo	428	394	41	356	1237	840	3296
Yuba	40	0	19	760	157	962	1939
Total by ai	5729	5433	12229	18254	9539	61602	112786

Source: DPR 2005.

Table 3: California Department of Food and Agriculture, Center for Analytical Chemistry analytical method details.

Organophosphate Insecticides in Surface Water by GC/FPD

Compound	Method Detection Limit (µg/L)	Reporting Limit (µg/L)
Azinphos methyl	0.0099	0.05
Chlorpyrifos	0.0008	0.01
Diazinon	0.0012	0.01
Dichlorvos	0.0098	0.05
Dimethoate	0.0079	0.04
Disulfoton	0.0093	0.04
Ethoprop	0.0098	0.05
Fenamiphos	0.0125	0.05
Fonofos	0.008	0.04
Malathion	0.0117	0.04
Methidathion	0.0111	0.05
Methyl Parathion	0.008	0.03
Phorate	0.0083	0.05
Profenofos	0.0114	0.05
Tribufos	0.0142	0.05

Pyrethroid Insecticides in Surface Water by GC/MSD

Compound	Method Detection Limit (ng/L)	Reporting Limit (ng/L)
Bifenthrin	2.16	5.0
Cyfluthrin	55.5	80.0
Cypermethrin	56.6	80.0
Esfenvalerate	22.5	50.0
Lambda-Cyhalothrin	7.76	20.0
Permethrin	16.9	50.0

Pyrethroid Insecticides in Sediment by GC/MSD 1

Compound	Method Detection Limit (μg/g)	Reporting Limit (µg/g)
Bifenthrin	See note 1	< 0.01
Cyfluthrin	< 0.008	< 0.01
Cypermethrin	< 0.008	< 0.01
Esfenvalerate	< 0.008	< 0.01
Lambda-Cyhalothrin	< 0.009	< 0.01
Permethrin	< 0.006	< 0.01

^{1.} Analytical limits for pyrethroids in sediment not yet available pending validation of new analytical method.

Figure 1. Average Pyrethroid Use, Monterey County, 2001 - 2003.

Note: Figures 1-4 depict agricultural use data for six pyrethroid active ingredients (permethrin, esfenvalerate, cyfluthrin, lambda-cyhalothrin, bifenthrin and cypermethrin), averaged over 2001-2003.

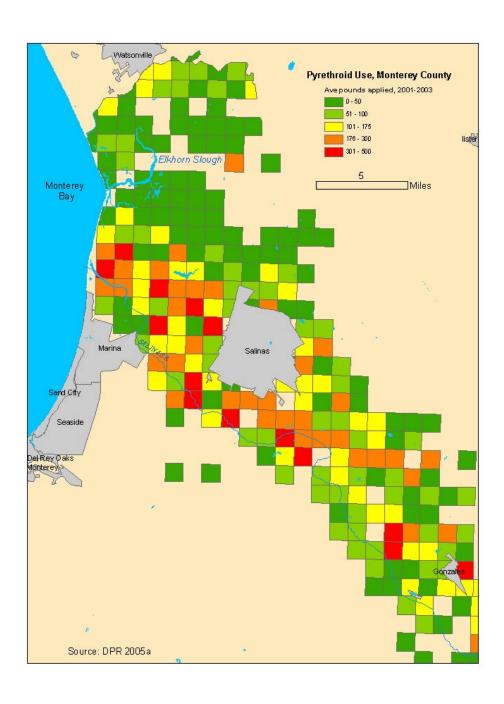


Figure 2. Average Pyrethroid Use, Imperial Valley, 2001-2003.

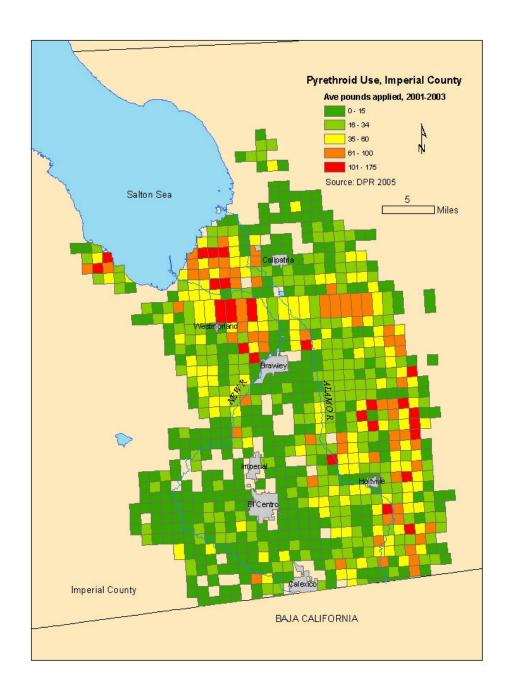


Figure 3. Average Pyrethroid Use, Sacramento Valley, 2001-2003.

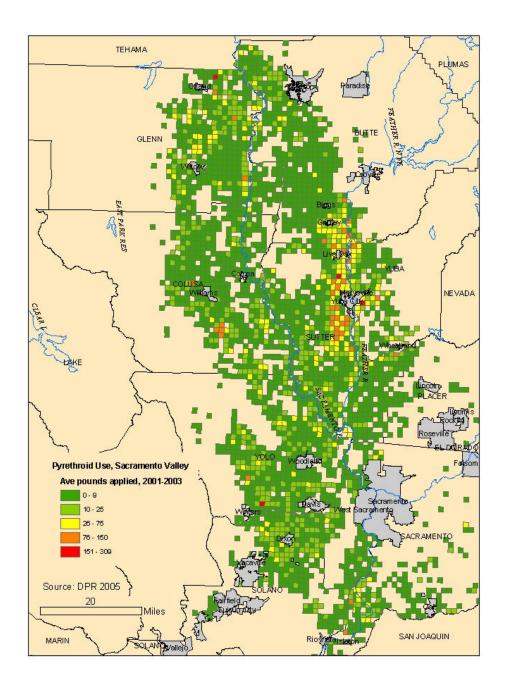


Figure 4. Average Pyrethroid Use, Northern San Joaquin Valley, 2001-2003.

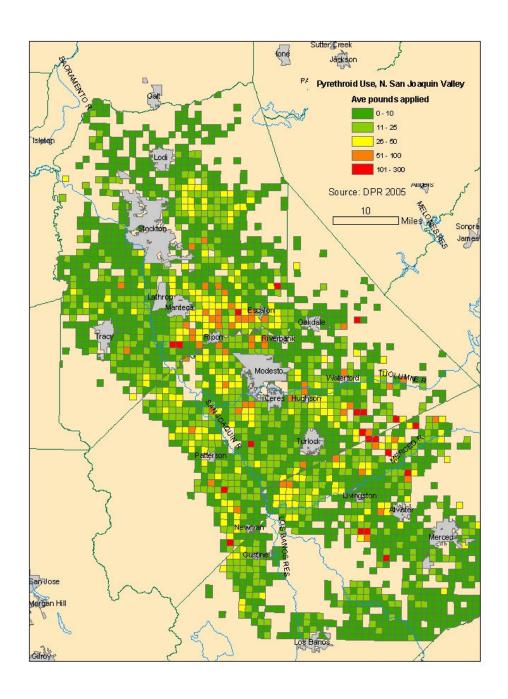
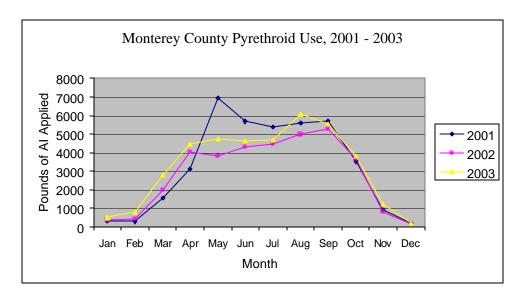


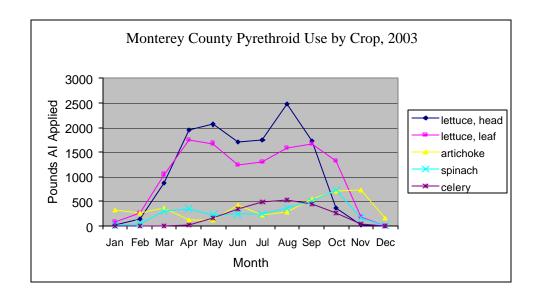
Figure 5. Monterey County Monthly Pyrethroid Use Data, 2001-2003.

All monthly use data are combined pounds of active ingredient for the six primary pyrethroid insecticides: permethrin, esfenvalerate, cyfluthrin, lambda-cyhalothrin, bifenthrin and cypermethrin. (DPR 2005a).



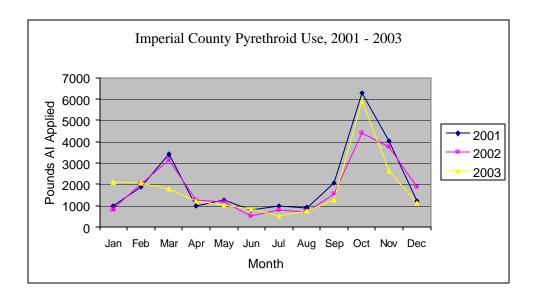
Monterey County monthly use data represents a use area of approximately 350 square miles.

Figure 6. Monterey County Pyrethroid Use by Crop, 2003



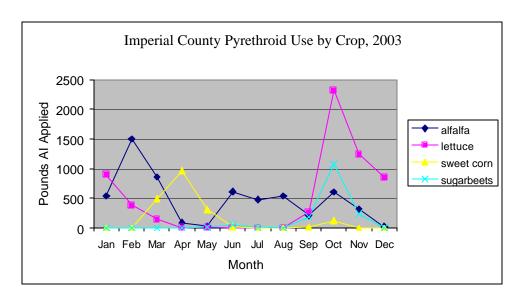
Crops depicted represent approximately 90% of all 2003 use.

Figure 7. Imperial County Monthly Pyrethroid Use Data, 2001-2003.



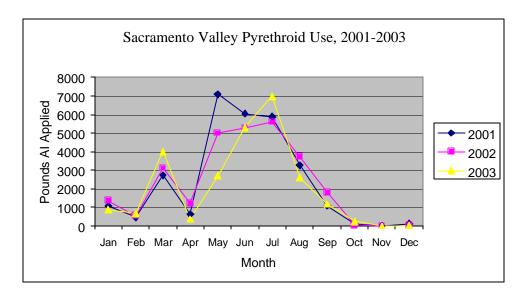
Imperial County monthly use data represents a use area of approximately 950 square miles.

Figure 8. Imperial County Pyrethroid Use by Crop, 2003.



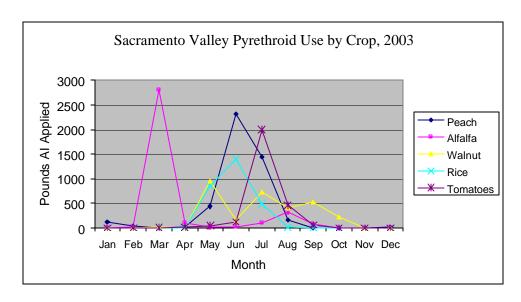
Crops depicted represent approximately 75% of all 2003 use.

Figure 9. Sacramento Valley Monthly Pyrethroid Use Data, 2001-2003.



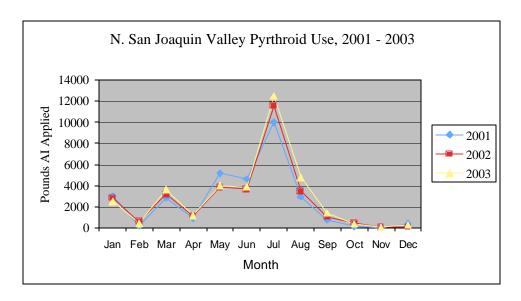
Sacramento Valley monthly use data represents a use area of approximately 3000 square miles.

Figure 10. Sacramento Valley Pyrethroid Use by Crop, 2003.



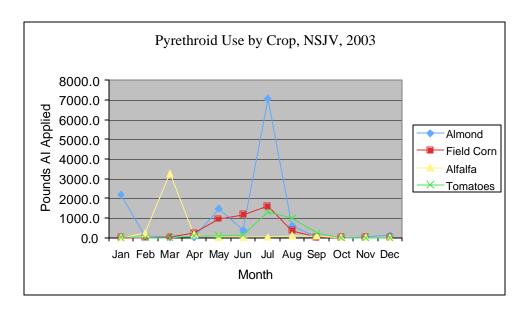
Crops depicted represent approximately 65% of all 2003 use.

Figure 11. Northern San Joaquin Valley Monthly Pyrethroid Use Data, 2001-2003.



Northern San Joaquin Valley monthly use data represents a use area of approximately 3000 square miles.

Figure 12. Northern San Joaquin Valley Pyrethroid Use by Crop, 2003.



Crops depicted represent approximately 65% of all 2003 use.